

Effect of APF Gel on the Micro Hardness of Sealant Materials

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Abstract: The possible adverse effects of topical fluoride treatment on restorative materials have been the subject of many studies during the last decades. Since, APF gels and sealant materials have many possibilities to interact in the oral cavity, it is necessary to understand such interaction. The aim of this study was to evaluate the effect of APF gel (Sultan) on the microhardness of a filled sealant material (Helioseal F) and an unfilled sealant material (Clinpro). A total of 16 cylindrical specimens (6 mm diameter and 2 mm thickness) from each material were fabricated and stored in distilled water at 37°C for 48 h. Half of the specimens were assigned as the control group and stored in distilled water for the corresponding periods of the fluoridation experiments. For the remaining half, the specimens were treated with 1.23% APF gel for 4 min. The application was followed by removing the gel and storing for 30 min in artificial saliva. Subsequently the specimens were immersed in distilled water for 30 min. The process of APF application was repeated. Vickers microhardness of each specimen was assessed and the results were analyzed using paired t-test ($p < 0.05$). Statistical analysis showed no statistically significant difference between the microhardness values of Helioseal F following APF treatment and controls (11.43 ± 2.35 vs. 12.79 ± 2.16). The microhardness of Clinpro was significantly reduced by exposure to APF gel (11.01 ± 0.89 vs. 12.95 ± 1.52). It seems filled sealants may be a better choice in respect to microhardness, when topical Acidulated Phosphate Fluoride (APF) gel is to be used routinely.

Key words: Restorative dentistry/dental material, prevention, APF gel, fluoride, sealant

INTRODUCTION

The sealant is used to protect the tooth surfaces most susceptible to dental caries. Many studies on the effectiveness of pit and fissure sealants have been performed. Currently, various types of pit and fissure sealants are available. Some are unfilled, while others contain various amounts of filler particle. The color of the sealant may be clear, tinted, or opaque. Some have fluoride as an active ingredient (Garcia-Godoy *et al.*, 2004). A major effort should be made to incorporate the use of sealant along with other primary preventive dentistry procedures, such as plaque control, fluoride therapy and sugar discipline (Garcia-Godoy *et al.*, 2004). The possible adverse effects of topical fluorides, particularly Acidulated Phosphate Fluoride (APF) application, on restorative materials have been documented in several studies (Kula *et al.*, 1992, 1983; Cehreli *et al.*, 2000; Soeno *et al.*, 2002). The composition and surface integrity of composites and other glass-containing restoratives can be significantly changed when exposed to strong acids

(El-Badrawy *et al.*, 1993; Papagiannoulis *et al.*, 1997; Diaz-Arnold *et al.*, 1995). This effect is of clinical significance because topical Acidulated Phosphate Fluoride (APF) gels, recommended as a preventive strategy for children and adolescents, contain strong acids (Soeno *et al.*, 2002).

These acids etch the enamel and consequently enhance the fluoride uptake (Brudevold *et al.*, 1963). Although, the effect of APF gel on composites has been widely reported, minimal literature regarding their effects on sealant materials is available.

For fissure sealants, which are frequently used in routine preventive treatments of children and adolescents, previous study by Kula *et al.* (1992) was based on morphologic assessment of the extent of surface roughness as observed by Scanning Electron Microscopy (SEM). Due to the potential softening, the effect of APF on microhardness of sealant materials also warrants investigation. This study investigated the effect of a commercial APF gel on the microhardness of filled and unfilled sealant materials.

MATERIALS AND METHODS

The materials selected for this study included a filled sealant material (Heliobond F) and an unfilled sealant material (Clinpro). Table 1 shows the technical profiles of the fissure sealant materials evaluated.

The fluoride gel used in the study was a 1.23%, Acidulated Phosphate Fluoride gel (APF) (Sultan APF, Sultan Health Care Inc., USA). For specimen preparation, plastic molds with dimensions of 6 mm in diameter by 2 mm in height were placed over a glass slide and fixed with wax. After material placement in the molds, a polyester strip (3 M ESPE dental products) and glass slide were placed over the material. Then, a weight of 100 g was applied over the glass plate for 30 sec. The specimens were polymerized for 40 sec side⁻¹ (Optilux 501, Sybron Kerr, USA). After curing, the cover glasses were removed and the plastic mold was separated. A total of 16 specimens were made for each material, which were further sub grouped into two groups (N = 8) serving as test and control specimens. The hardened specimens were stored in distilled water for 48 h at 37°C.

Each test specimen was immersed in 1.23% APF gel and left in place for 4 min. The gel was wiped off with cotton rolls and each specimen was placed in 2 mL of artificial saliva (0.003 M NaH₂PO₄, 0.02 M NaHCO₃, 0.001 M CaCl₂, pH = 7.0) and stored at 37°C for 30 min. Subsequently, the specimens were immersed in distilled water at 37°C for 30 min. The process of APF application and storing in artificial saliva was repeated for each specimen. The controls (N = 8 for each material) were subjected to the same regimen, except they were stored in distilled water for the corresponding periods of the fluoridation experiments. Upon completion of the treatment, all specimens were washed, dried and subjected to microhardness testing with a load of 10 g applied for 10 sec using a microhardness tester (Adolph Beuhler Inc., Lake Bluff, III, USA) at the top surface. Three readings were taken for each specimen and averaged to form a single value for each specimen.

Paired t-test was used to assess the statistically significant differences in microhardness between the controls and APF treated at a significant level of $p < 0.05$. The statistical analysis was carried out with SPSS 11 software.

Table 1: Technical profiles of the fissure sealant materials evaluated

Material	Manufacturer	Composition
Heliobond F	Ivoclar North America	Bis-GMA, UDMA, TEGDMA, silicon dioxide and fluorosilicate glass (40.5% by weight), releases fluoride
Clinpro	3 M ESPE	Bis-GMA, TEGDMA, unfilled, releases fluoride
Bis-GMA: Bisphenol Glycidyl Methacrylate; UDMA: Urethane Dimethacrylate; TEGDMA: Triethyleneglycol Dimethacrylate		

Table 2: Mean and standard deviations of the Vicker's microhardness values of the groups tested

Materials	Control (N = 8)	APF treated (N = 8)
Heliobond F	12.79±2.16	11.43±2.35
Clinpro	12.95±1.52	11.01±0.89

RESULTS

Table 2 presents the mean microhardness values including standard deviations of the two sealant materials investigated after distilled water storage (controls) and storing in APF gel. The results of each group were evaluated by paired t-test. The sealant material Clinpro presented a statistically significant decrease in microhardness after the APF treatment compared with its control group ($p < 0.05$). For the sealant material Heliobond, no significant difference in microhardness values was observed after APF treatment as compared with the control group ($p > 0.2$).

DISCUSSION

Hardness may be defined as the resistance of a material to indentation or penetration (O'Brien, 2002). However, it is difficult to formulate a definition that is completely acceptable, as the indentation produced results from the interaction of numerous properties. Among the properties related to the hardness of a material are strength, proportional limit and its ability to abrade or be abraded by opposing dental structures and materials (Anusavice, 1996). Material loss in the pit and fissures is thought to be the key factor in caries preventive function of pit and fissure sealant (Kim *et al.*, 2002). Therefore, any hardness deterioration observed with the use of professionally applied topical fluorides has implications on the clinical durability of these restorations (Yap and Mok, 2002). The significance of APF treatment on surface microhardness of sealant materials, however, has not yet been investigated. Therefore, it was the aim of this study to elucidate if microhardness of these materials decreases after APF treatment. Previous investigations have shown the suitability and practicality of using Vicker's microhardness test for evaluating the surface changes of pit and fissure sealants (Kim *et al.*, 2002).

In the present study, the hardened materials were stored for 48 h in distilled water at 37°C prior to fluoride treatment to allow for post-polymerization, which affects hardness reading (Garcia-Godoy *et al.*, 2003). Increasing the degree of cure of matrix polymer will inhibit diffusion of penetrants and the additional cross-linking will reduce swelling and damage by solvents (McKinney and Wu, 1985). The fluoride treatment regimen (twice for 4 min at

37°C) employed was based upon a commonly biannual application of APF. According to Donly and Stookey (2004), whichever fluoride system is used for topical fluoride applications, the teeth should be exposed to the fluoride for 4 min for maximal cariostatic benefits. Subsequently, the gel was removed and each specimen was stored in artificial saliva at 37°C for 30 min that simulates 30 min of contact prior to eating, drinking or rinsing (Donly and Stookey, 2004). Although, this does not represent the long-term clinical effect, it reflects the immediate potential effect of the use of the gel (Garcia-Godoy *et al.*, 2003). Afterwards, the specimens were immersed in distilled water at 37°C for 30 min to prevent chemical equilibrium formation at the material surface and to provide an intermediate relaxation period (Papagiannoulis *et al.*, 1997).

Three major interaction pathways among the materials and fluoride agents may be identified as described by Papagiannoulis *et al.* (1997). Interactions exist with organic matrix, filler-matrix coupling agents and/or reinforcing fillers. The organic matrixes of the sealant materials evaluated are both organic esters of methyl methacrylate derivatives. Organic esters undergo hydrolytic cleavage of the ester group in low pH. This reaction is acid-catalyzed and is pH-dependent (Yap and Mok, 2002). As APF gel is acidic, the amount of water bound to organic matrixes is increased (Papagiannoulis *et al.*, 1997), resulting in the decreased hardness that is observed. Significant differences in hardness between specimens treated with APF gel and controls, however, only observed with the unfilled sealant (Clinpro). For the filled sealant (Helioclear F), no significant difference in hardness was observed between treatment with APF gel and distilled water. A maximum softening effect is expected when the value of the magnitude of solubility parameter of a liquid is equal to that of the matrix polymer of the composite (McKinney and Wu, 1985). Wu and McKinney (1982) reported that the Bis-GMA resin polymer can be softened by chemicals with solubility parameters in the range of 18.2-29.7 (Mpa)^{1/2}. Helioclear F uses a polymer base slightly different from that of the Clinpro (Table 1) and therefore, may be less sensitive to APF gel. APF Sultan contains a variety of chemical agents. It is unclear whether any of these agents have solubility parameters similar to that of polymer base of Clinpro. Furthermore, the surface changes could be caused by complex interactions within this multicomponent APF product, rather than by one specific chemical component.

The decreased hardness after treatment with APF gel could also attributed to the presence of hydrofluoric acid

(Cehreli *et al.*, 2000). Hydrogen ions from the phosphoric acid and fluoride ions from sodium fluoride are present in the APF solution and hydrofluoric acid is therefore, generated (Soeno *et al.*, 2001). Hydrofluoric acid dissolves composite filler particles that contribute to decrease in surface hardness. The effect of APF agents on resin composites appears to be related to the type of filler particles in the resin composite (Papagiannoulis *et al.*, 1997; Kula *et al.*, 1986). Resin composites containing baroalumino silicate glass particles are among the most susceptible to surface changes caused by APF. The lack of statistically decreased microhardness after APF treatment of Helioclear F is thought to be because of the fact that no wear occurs when the filler particles were very closely spaced. Jorgensen and Asmussen (1978) postulated that if the inter particle separation between high modulus filler particles were <0.1 µm, then perhaps wear would be precluded by the protection of the matrix by the neighboring filler particles. However, this study cannot confirm the exact mechanism and warrants further investigation. In contrast to the present study, the majority of the studies evaluating the effect of fluoride agents used only filler-based restorative materials.

Given the degree of the microhardness reduction caused by gels, care should be taken in the selection of sealant material where, APF application is likely. Accordingly, it is desirable to select filled sealant materials, which is less influenced by APF. Clinically, the effects of APF gel on fissure sealant materials, however, may be modified by many factors that could not be replicated in this study. Saliva may dilute or buffer the gel. Also, in the oral environment the presence of salivary proteins may in some way protect the material surface immediately before and after the gel application (El-Badrawy *et al.*, 1993). The results reported might therefore, exaggerate the effect that APF gels have on these materials. Therefore, longitudinal clinical studies should be carried out in order to evaluate the long-term performance of these materials after exposure to APF gels and therefore, support the findings of the present *in vitro* study.

CONCLUSION

The results of this *in vitro* study suggest that APF gel alters the surface hardness of unfilled sealant materials and in respect to microhardness, it is preferable to use filled sealants if patients are to receive oral care including APF application to reduce any adverse effect.

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